

0010457

ERT

ENVIRONMENTAL RESEARCH & TECHNOLOGY INC

ADMINISTRATIVE RECORD

SF FILE NUMBER

401

MEMORANDUM

TO: John Ryan

MEMO NO.: D236-110

FROM: Gary Wilson *Gary Wilson*

FILE:

SUBJECT: Critique of analytical results for PAH at ppt levels in water - BNRR

DATE: September 17, 1984

Over the summer ERT's analytical laboratory analyzed 75-100 water samples for PAH at parts per trillion (ppt) levels. This memo discusses specific problems encountered, how the problems were dealt with, and future plans. We are currently developing a job specific PAH analytical and QC protocol to be used on subsequent BNRR work. The analytical methods, of course, were already developed, but we are making adjustments which should yield better results for the BNRR sample matrix. The QC protocol is being developed from the QA/QC sample data generated during the work this summer.

Specific Problems

1) Ultra-trace level organic analysis is inherently more variable than most environmental analysis work. PAH's by their nature complicate the problem. When analyzing PAH at part per trillion levels we are looking at 10^{-8} to 10^{-9} grams per millileter. PAH easily adsorb on solids. With only picogram (10^{-9} gram) quantities of PAH involved, a very small number of adsorption sights can cause a loss which causes a large difference in the final result. To avoid this problem, glassware is baked at high temperature after a thorough cleaning. However, the required handling of the sample during preparation and analysis results in potential losses which can not be completely eliminated. For this reason, reported confidence limits on recovery of PAH from water are highly variable. Our analytical work has shown similar variability.

This is a problem which can not be "corrected". We will continue to define the variability of ultra-trace analyses performed in the Concord laboratory so that our data can be interpreted correctly. Table I shows the ERT PAH recovery data averages and the EPA confidence limits for reference laboratory analysis of EPA spikes. It should be noted that the EPA data was generated for part per billion levels, not part per trillion levels, and therefore the variability of the EPA data should be better than achievable at part per trillion levels.

The statistical calculation of a 95% confidence interval is a means of developing action criteria. The 95% confidence interval says that statistically 95% of all recovery values will fall within the limits given.

Page 2

These upper and lower concentration limits act as control limits. Ultimately, the control limits can be used to define the efficiency of the analysis.

2) A second problem with part per trillion measurement is various sources of background contamination. ERT performs analysis of blank distilled water to determine, for each analysis batch, the amount of field and laboratory background contamination (if any). Samples are corrected by subtracting the laboratory background concentration. However, variation in the background concentration affects the detection limit for each compound. Even though the instrument detection limit may remain constant, variations in background concentration between compounds and between batches causes the final reportable detection limit to change.

ERT takes extreme precautions to avoid as much background contamination as possible. However at part per trillion sensitivity it is impossible to totally eliminate background of these commonly occurring compounds. Therefore, ERT performs sufficient blank analyses to obtain enough data to statistically determine the level and variability of the background contamination.

3) A third problem encountered during this summer analyses was a recurring high molecular weight contamination which interfered with the analysis of mass 252 PAH (benzofluoranthenes and benzo (a) pyrene). Even with background correction, the identification of mass 252 was very complicated. In such a situation, on a questionable identification we must quantitate as if the sample contained the PAH. In order to avoid this problem in the future, we are making several minor adjustments to the analytical method. These include a pH adjustment of the samples prior to extraction and use of a column cleanup procedure. By eliminating the interference from the extract, we will increase our ability to reliably confirm the presence or absence of the mass 252 PAH, consistent with our confirmation for the other PAH compounds measured.

0010459

TABLE I: ANALYTICAL RECOVERYS
% RECOVERY

	MEAN	STANDARD DEVIATION	95% CONF. LIMITS ERT	EPA
Naphthalene	77	51	43-112	8-104
Acenaphthylene	66	27	50-83	9-123
Acenaphthene	84	20	73-96	BDL-110
Phenanthrene/Anthracene	40	22	27-53	6-129
Fluoranthene	105	32	87-124	24-111
Pyrene	105	25	91-120	11-125
Benz(a)Anthracene/ Chrysene	81	43	45-117	18-143
Benzofluoranthenes	96	58	55-138	12-139
Benzo(a)pyrene	88	54	47-130	BDL-115
Dibenz(ah)Anthracene	25	27	BDL-59	8-128
Benzo(ghi)perylene	28	21	BDL-60	7-135